B. Selected Technical Reports

1. *Development of a Neutron Incoherent Scattering Method to Quantify Hydrogen in Metals*

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Objective: To develop and critically evaluate Neutron Incoherent Scattering for the determination of hydrogen in metals.

Problem: It is well known that hydrogen causes embrittlement in metals. Methods used in industry for determining H in metals are highly matrix dependent. Therefore, the metals industry requires SRMs containing H in a variety of alloys to calibrate and verify laboratory methods. Although we have one method based on PGAA for non-destructive H determination, a second method is highly desirable for confirming analytical results. The new measurement method is based on the incoherent scattering of neutrons by the H nucleus, which differs from PGAA in that the latter relies on the absorption of a neutron and subsequent emission of a 2.2 MeV gamma ray by H.

Approach: The incoherent scattering cross section of neutrons by the H nucleus is much greater than both the scattering cross section of any other nucleus and the absorption cross section of H. These two properties of the neutron interaction with H establish the potential of using neutron incoherent scattering (NIS) for H determination with higher efficiency and better sensitivity than PGAA. Since measurements can be made in minutes, compared to hours for PGAA, the NIS technique can be used effectively to study homogeneity. PGAA, however, is much more selective and matrix independent. The two techniques have a very limited overlap of error sources, and therefore, agreement of results between the two techniques provides a high degree of confidence.

A good understanding of the neutron scattering mechanism is needed for each type of sample analyzed by NIS. The value of the scattering cross section depends on the binding state of H, i.e., whether it behaves more like a free gas (scattering is in the forward direction), or is rigidly bound to the matrix (scattering is isotropic). We have built a test system to measure scattering in both forward and backward directions to determine which of these mechanisms is appropriate. Using standard addition techniques and making the assumption that H is rigidly bound in metals, we have obtained quantitative results that are in agreement with PGAA measurements.

Results and Future Plans: We have determined H concentrations by NIS measurements in a series of titanium alloys (45-350 µg-H/g-Ti), and compared the results with those obtained by PGAA. All values are in agreement with the exception of one sample at 300 µg/g. Further investigation is required to understand this discrepancy. We have studied H loading dynamics in a rare-earth window coating material used for reversible opacity control. The window opacity is altered by varying the hydrogen gas pressure which in turn affects the subsequent hydride formation within the material. When conditions are varying, a fast probe like NIS is indispensable. PGAA is not an appropriate technique for these measurements since it can only provide quantitative information when the system is in a steady state. We are currently testing a series of samples containing approximately 100 µg-H/g-Ti to be used as a future SRM. The NIS method's advantage of rapid determination allows scanning measurements of large metal sheets to obtain information on concentration uniformity. Currently, the experimental station is time-shared with other measurement applications, and each time the equipment is changed a complete background reduction effort and a new calibration is required. We intend to build a permanent chamber with neutron-black walls and a high precision sample-positioning device for the NIS measurement, with which the critical evaluation of the technique can be completed.